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S/N 09/614,631

PATENTIN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: HILGREN ET AL. Examiner: J. PAK  
Serial No.: 09/614,631 Group Art Unit: 1653  
Filed: JULY 12, 2000 Docket No.: 163.1382US01  
Title: METHOD AND COMPOSITION FOR INHIBITION OF MICROBIAL  
GROWTH IN AQUEOUS FOOD TRANSPORT AND PROCESS  
STREAMS

<b>CERTIFICATE UNDER 37 CFR 1.601:</b> I hereby certify that this paper is being transmitted by facsimile to the U.S. Patent and Trademark Office on <u>August 5</u> , 2005.	
By:	<i>Sheryl A. Boerboom</i>
Name:	<i>Sheryl A. Boerboom</i>

DECLARATION UNDER 37 CFR § 1.131

Commissioner for Patents  
Washington, D.C. 20231

Dear Sir:

I, John D. Hilgren, declare and state the following:

1. I am an inventor of the subject matter of the patent application identified above and an employee of Ecolab, Inc., the assignee of the patent application identified above.
2. I understand that the Examiner has cited Hei (US 6,024,986) as prior art in prosecution of the application identified above. I understand that the Hei patent was filed on May 24, 1999.
3. I further understand that the filing date of the present patent application Serial No. 09/614,631 is July 12, 2000.
4. I state that before the date of the Hei patent, before May 24, 1999, my coinventors and I invented the subject matter described and claimed in the patent application identified above.
5. The claims of the patent application identified above relate to compositions including:

about 35 to about 45 weight-% acetic acid, about 5 to about 15 weight-% octanoic acid, about 3 to about 8 weight-% hydrogen peroxide, about 8 to about 16 weight-% peroxyacetic acid, about 1 to about 5 weight-% peroxyoctanoic acid, and about 0.1 to about 2 weight-% chelating agent;

about 40 weight-% acetic acid, about 10 weight-% octanoic acid, about 5 weight-% hydrogen peroxide, about 12 weight-% peroxyacetic acid, about 3 weight-% peroxyoctanoic acid, and about 0.6 weight-% chelating agent;

about 10 to about 150 ppm acetic acid, about 5 to about 40 ppm octanoic acid, about 4 to about 20 ppm hydrogen peroxide, about 5 to about 50 ppm peroxyacetic acid, about 2 to about 25 ppm peroxyoctanoic acid, and about 0.2 to about 2.5 ppm chelating agent;

about 133 ppm acetic acid, about 33 ppm octanoic acid, about 17 ppm hydrogen peroxide, about 40 ppm peroxyacetic acid, about 10 ppm peroxyoctanoic acid, and about 2 ppm chelating agent;

about 50 to about 60 weight-% acetic acid, about 10 to about 20 weight-% octanoic acid, about 5 to about 15 weight-% hydrogen peroxide, and about 0.3 to about 1 weight-% chelating agent; or

about 54 weight-% acetic acid, about 14 weight-% octanoic acid, about 10 weight-% hydrogen peroxide, and about 0.6 weight-% chelating agent;

Each claimed composition also has at least about 1 part by weight of peroxyoctanoic acid for each about 5 parts of peroxyacetic acid.

The present patent application includes at page 15 several tables describing embodiments of the claimed compositions. Two of the tables describe concentrate and use compositions including:

Chemical	Wt-%	ppm
Acetic Acid	40	133
Hydrogen Peroxide	5	17
HEDP	0.6	2
Octanoic Acid	10	33
Peroxyacetic Acid	12	40
Peroxyoctanoic Acid	3	10

One of the tables describes raw materials that can be used to make the concentrate composition described above. These raw materials include:

Raw Material	Weight %
Glacial Acetic Acid	54
Hydrogen Peroxide, 35%	30
HEDP, 60%	1
Octanoic Acid, 95%	15

6. During the research and development leading to the claimed compositions, and before May 24, 1999, my coinventors and I made several compositions meeting the limitations of the claims of the present application and including the compositions described in the tables in paragraph 5 above.

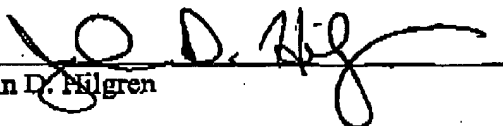
7. Accompanying Exhibit A describes several use compositions that meet the ratio limitation of the present claims and that can be made from concentrates according to the present invention. These include Formulas B, C, D, and E. The contents of these formulas can be found at page 2 of Exhibit A. Page 3 of Exhibit A describes the antimicrobial activity of these formulas. The report included as Exhibit A was prepared before May 24, 1999.

8. Accompanying Exhibit B reports studies of compositions that meet the limitations of the present claims and including the compositions described in the Tables in paragraph 5 above. In Exhibit B, the initial, concentrate, and use compositions described in the tables in paragraph 5 above are referred to as "Falcon 15 O". The composition called "Falcon 15 AE" also meets the limitations of the present claims. The contents of these formulas can be found at page 2 of Exhibit B. Pages 3-4 of Exhibit B describe the antimicrobial activity of these formulas. The report included as Exhibit B was prepared before May 24, 1999.

9. The evidence presented in Exhibits A and B indicates that, before May 24, 1999, my coinventors and I invented the subject matter described and claimed in the patent application identified above.

10. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States

Code, and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

  
John D. Hilgren

**EXHIBIT A  
ATTACHED TO  
DECLARATION UNDER 37 CFR § 1.131  
U.S. PATENT SERIAL NO. 09/614,631**

# ECOLAB

Ecolab Inc.  
Microbiological Services  
840 Sibley Memorial Highway  
Mendota Heights, Minnesota 55118  
651-306-5600

## MICROBIOLOGICAL SERVICES REPORT

Rate of Kill of Eight New Formulations to Examine the Effects  
of POOA, OA and NAS Against an *Aspergillus* sp. Isolate

MS.R. #

SUBMITTED BY

John Hilgren, Food and Beverage Division

DATE SUBMITTED

REPORTED BY

COMPLETION DATE

Ex  
A



Accredited by the Council for Antimicrobial Quality in Phase I of its Laboratory Accreditation Program for GLP compliance and data integrity

**OBJECTIVE:**

The objective of the analysis was to determine the rate of kill of eight new formulations when tested at various concentrations of POOA, OA and NAS against an *Aspergillus* sp. field isolate.

**TEST METHOD:**

Ecolab Microbiological Services SOP

; Rate of Kill Antimicrobial Efficacy

**METHOD PARAMETERS:**

Test Substance Name	Concentration	Pre-test pH
Formula A	80 ppm POAA, 16 ppm H <sub>2</sub> O <sub>2</sub> , 133 ppm Acetic Acid, 3 ppm POOA, 10 ppm OA	3.71
Formula B	80 ppm POAA, 16 ppm H <sub>2</sub> O <sub>2</sub> , 133 ppm Acetic Acid, 17 ppm POOA, 10 ppm OA	3.72
Formula C	80 ppm POAA, 16 ppm H <sub>2</sub> O <sub>2</sub> , 133 ppm Acetic Acid, 17 ppm POOA, 91 ppm OA	3.66
Formula D	80 ppm POAA, 16 ppm H <sub>2</sub> O <sub>2</sub> , 133 ppm Acetic Acid, 17 ppm POOA, 91 ppm OA, 116 ppm NAS	3.67
Formula E	80 ppm POAA, 16 ppm H <sub>2</sub> O <sub>2</sub> , 133 ppm Acetic Acid, 17 ppm POOA, 10 ppm OA, 116 ppm NAS	3.71
Formula F	80 ppm POAA, 16 ppm H <sub>2</sub> O <sub>2</sub> , 133 ppm Acetic Acid, 3 ppm POOA, 91 ppm OA	3.69
Formula G	80 ppm POAA, 16 ppm H <sub>2</sub> O <sub>2</sub> , 133 ppm Acetic Acid, 3 ppm POOA, 10 ppm OA, 116 ppm NAS	3.72
Formula H	80 ppm POAA, 16 ppm H <sub>2</sub> O <sub>2</sub> , 133 ppm Acetic Acid, 3 ppm POOA, 91 ppm OA, 116 ppm NAS	3.68

Diluent: Sterile Distilled Water

Test System: *Aspergillus* sp. (Gilroy Foods Isolate)

Test Temperature: 25°C

Exposure Times: 10, 20, 30 and 40 minutes

Neutralizer: 1% Sodium Thiosulfate

Subculture Medium: Sabouraud Dextrose Agar

Incubation: 26°C for 48 hours

**RESULTS:**

Test Date:

Date Results Read:

**Inoculum Numbers (CFU/mL)**

Test System	A	B	C	Average
<i>Aspergillus</i> sp. (Gilroy Foods Isolate)	8 x 10 <sup>5</sup>	9 x 10 <sup>5</sup>	8 x 10 <sup>5</sup>	8.3 x 10 <sup>5</sup>

*Aspergillus sp.*

Test Substance	Post-test pH	Exposure Time	Survivors (CFU/mL)	Average Survivors (CFU/mL)	Log Reduction	Percent Reduction
Formula A	4.02	10 minutes	$19, 26 \times 10^5$	$2.2 \times 10^6$	None	None
		20 minutes	$11, 13 \times 10^5$	$1.2 \times 10^6$	None	None
		30 minutes	$8, 12 \times 10^5$	$1.0 \times 10^6$	None	None
		40 minutes	$7, 6 \times 10^5$	$6.5 \times 10^5$	0.11	21.687
Formula B	3.74	10 minutes	$39 \times 10^3, <10$	$2.0 \times 10^4$	1.54	97.590
		20 minutes	$8, 32 \times 10^3$	$2.0 \times 10^4$	1.54	97.590
		30 minutes	$60 \times 10^1, 6 \times 10^3$	$3.3 \times 10^3$	2.40	99.602
		40 minutes	$28, 28 \times 10^3$	$2.8 \times 10^4$	1.47	96.626
Formula C	3.69	10 minutes	$11, 40 \times 10^3$	$2.6 \times 10^4$	1.50	96.867
		20 minutes	$3, 3 \times 10^3$	$3.0 \times 10^3$	2.44	99.638
		30 minutes	$8 \times 10^1, 3 \times 10^3$	$1.5 \times 10^3$	2.74	99.819
		40 minutes	$2 \times 10^1, 1 \times 10^3$	$5.1 \times 10^2$	3.21	99.938
Formula D	3.71	10 minutes	$32, 27 \times 10^3$	$3.0 \times 10^4$	2.44	96.386
		20 minutes	$52 \times 10^1, 5 \times 10^3$	$2.8 \times 10^3$	2.47	99.663
		30 minutes	$20 \times 10^1, 2 \times 10^3$	$1.1 \times 10^3$	2.88	99.867
		40 minutes	$1 \times 10^3, 12 \times 10^1$	$5.6 \times 10^2$	3.17	99.932
Formula E	3.74	10 minutes	$54, 66 \times 10^3$	$6.0 \times 10^4$	1.14	92.771
		20 minutes	$21, 42 \times 10^3$	$3.2 \times 10^4$	1.41	96.144
		30 minutes	$59 \times 10^1, 15 \times 10^3$	$7.8 \times 10^3$	2.03	99.060
		40 minutes	$12 \times 10^3, 49 \times 10^1$	$6.2 \times 10^3$	2.13	99.253
Formula F	3.71	10 minutes	$7, 17 \times 10^5$	$1.2 \times 10^6$	None	None
		20 minutes	$9, 10 \times 10^5$	$9.5 \times 10^5$	None	None
		30 minutes	$67, 68 \times 10^4$	$6.8 \times 10^4$	1.09	91.807
		40 minutes	$79, 68 \times 10^3$	$7.4 \times 10^4$	1.05	91.084
Formula G	3.77	10 minutes	$32, 16 \times 10^5$	$2.4 \times 10^6$	None	None
		20 minutes	$11, 14 \times 10^5$	$1.3 \times 10^6$	None	None
		30 minutes	$11, 11 \times 10^5$	$1.1 \times 10^6$	None	None
		40 minutes	$9, 17 \times 10^5$	$1.3 \times 10^6$	None	None
Formula H	3.70	10 minutes	$12, 20 \times 10^5$	$1.6 \times 10^6$	None	None
		20 minutes	$8, 14 \times 10^5$	$1.1 \times 10^6$	None	None
		30 minutes	$70, 109 \times 10^3$	$9.0 \times 10^4$	0.96	89.157
		40 minutes	$55, 79 \times 10^3$	$6.7 \times 10^4$	1.09	91.928



**CONCLUSIONS:**

Formulas C and D were the most effective, achieving a >99.9% reduction of *Aspergillus* sp. after a 40 minute exposure time. The surfactant (NAS) did not appear to be a significant contributing factor in increased efficacy but the peroxyoctanoic acid (POOA) and the octanoic acid (OA) appeared to be the contributing factors in increased efficacy.

Tested and Certified by:

\_\_\_\_\_  
Date

**EXHIBIT B  
ATTACHED TO  
DECLARATION UNDER 37 CFR § 1.131  
U.S. PATENT SERIAL NO. 09/614,631**

**MICROBIOLOGICAL SERVICES REPORT**

**Food Contact Surface Sanitizing Efficacy of Falcon 15 AE, Falcon 15 O and KX-6049  
Against *Staphylococcus aureus* ATCC 6538, *Escherichia coli* ATCC 11229 and  
*Saccharomyces cerevisiae* ATCC 834 at 25°C**

**M.S.R. #**

Ex

**SUBMITTED BY**

B

**DATE SUBMITTED**

**REPORTED BY**

**COMPLETION DATE**

**OBJECTIVE:**

The objective of the analysis was to determine the food contact surface sanitizing efficacy of Falcon 15 AE, Falcon 15 O and KX-6049 against *Staphylococcus aureus* ATCC 6538, *Escherichia coli* ATCC 11229 and *Saccharomyces cerevisiae* ATCC 834 at 25°C and 25 ppm POAA.

**TEST METHOD:**

Ecolab Microbiological Services SOP

*Germicidal and Detergent Sanitizing Action of Disinfectants - Final Action*

**METHOD PARAMETERS:**

- Test Substances:
1. Falcon 15 AE
  2. Falcon 15 O
  3. KX-6049

**Chemical Properties of Each Test Formula**

Formula	ppm POAA	ppm H <sub>2</sub> O <sub>2</sub>	ppm AA	ppm POOA	ppm OA
Falcon 15 AE	25	10	130	4	19
Falcon 15 O	25	10	109	6	23
KX-6049	25	50	231	3	31

Test Substance Name	Diluent	Concentration ppm (v/v)	mL of Test Substance	mL of Diluent	Pre-test pH
Falcon 15 AE	Sterile Distilled Water	257 ppm	0.26	999.74	3.73
Falcon 15 O	Sterile Distilled Water	241 ppm	0.24	999.76	3.78
KX-6049	Sterile Distilled Water	824 ppm	0.82	999.18	3.54

Test Systems: *Staphylococcus aureus* ATCC 6538  
*Escherichia coli* ATCC 11229  
*Saccharomyces cerevisiae* ATCC 834

Test Temperature: 25°C

Exposure Times: 10 seconds  
 20 seconds  
 30 seconds

Neutralizer: 1.0% Sodium thiosulfate

Subculture Media: Tryptone Glucose Extract (TGE) Agar for *S. aureus* & *E. coli*  
 Sabouraud Dextrose (SAB) Agar for *S. cerevisiae*

Incubation: 37°C for 48 hours (*S. aureus* & *E. coli*)  
 26°C for 72 hours (*S. cerevisiae*)

**RESULTS:**

Test Date:

Date Results Read:

**Inoculum Numbers (CFU/mL)**

Organism	A	B	C	Average
<i>S. aureus</i>	$29 \times 10^6$	$45 \times 10^6$	$46 \times 10^6$	$4.0 \times 10^7$
<i>E. coli</i>	$99 \times 10^6$	$104 \times 10^6$	$87 \times 10^6$	$9.7 \times 10^7$
<i>S. cerevisiae</i>	$15 \times 10^5$	$21 \times 10^5$	$12 \times 10^5$	$1.6 \times 10^6$

***Staphylococcus aureus* ATCC 6538**

Test Substance	Exposure Time (seconds)	Survivors (CFU/mL)	Average Survivors (CFU/mL)	Log Reduction	Post-test pH
Falcon 15 AE	10	$10, 5 \times 10^1$	$7.5 \times 10^1$	5.73	3.78
	20	<10, <10	$<1.0 \times 10^1$	>6.60	
	30	<10, <10	$<1.0 \times 10^1$	>6.60	
Falcon 15 O	10	<10, <10	$<1.0 \times 10^1$	>6.60	3.84
	20	<10, <10	$<1.0 \times 10^1$	>6.60	
	30	<10, <10	$<1.0 \times 10^1$	>6.60	
KK-6049	10	$10, 15 \times 10^1$	$1.3 \times 10^2$	5.49	3.59
	20	<10, <10	$<1.0 \times 10^1$	>6.60	
	30	<10, <10	$<1.0 \times 10^1$	>6.60	

***Escherichia coli* ATCC 11229**

Test Substance	Exposure Time (seconds)	Survivors (CFU/mL)	Average Survivors (CFU/mL)	Log Reduction	Post-test pH
Falcon 15 AE	10	$13.24 \times 10^3$	$1.9 \times 10^4$	3.71	3.79
	20	<10, <10	$<1.0 \times 10^1$	>6.99	
	30	<10, <10	$<1.0 \times 10^1$	>6.99	
Falcon 15 O	10	$65.38 \times 10^1$	$5.2 \times 10^2$	5.27	3.85
	20	<10, <10	$<1.0 \times 10^1$	>6.99	
	30	<10, <10	$<1.0 \times 10^1$	>6.99	
KX-6049	10	$3.1 \times 10^3$	$2.0 \times 10^3$	2.69	3.59
	20	<10, <10	$<1.0 \times 10^1$	>6.99	
	30	<10, <10	$<1.0 \times 10^1$	>6.99	

***Saccharomyces cerevisiae* ATCC 834**

Test Substance	Exposure Time (seconds)	Survivors (CFU/mL)	Average Survivors (CFU/mL)	Log Reduction	Post-test pH
Falcon 15 AE	10	$3.1 \times 10^5$	$2.0 \times 10^5$	0.90	3.81
	20	<10, <10	$<1.0 \times 10^1$	>5.20	
	30	<10, <10	$<1.0 \times 10^1$	>5.20	
Falcon 15 O	10	$2.3 \times 10^4$	$2.5 \times 10^4$	4.81	3.85
	20	<10, <10	$<1.0 \times 10^1$	>5.20	
	30	<10, <10	$<1.0 \times 10^1$	>5.20	
KX-6049	10	$5.6 \times 10^5$	$5.5 \times 10^5$	0.46	3.60
	20	$12.14 \times 10^3$	$1.3 \times 10^4$	2.09	
	30	<10, <10	$<1.0 \times 10^1$	>5.20	

**CONCLUSIONS:**

Falcon 15 AE, Falcon 15 O and KX-6049 all achieved a >5.00 log reduction after a 10 second exposure time against *Staphylococcus aureus* ATCC 6538 at 25°C. Falcon 15 O demonstrated the best efficacy against *Escherichia coli* ATCC 11229 by achieving a >5.00 log reduction after a 10 second exposure time, while Falcon 15 AE and KX-6049 achieved a >5.00 log reduction after 20 seconds. Falcon 15 O also demonstrated the best efficacy against *Saccharomyces cerevisiae* ATCC 834 by achieving a 4.81 log reduction after a 10 second exposure time and a >5.00 log reduction after a 20 second exposure time. Falcon 15 AE demonstrated similar efficacy by achieving a >5.00 log reduction after a 20 minute exposure time while achieving a <1.00 log reduction after a 10 second exposure time. KX-6049 achieved a >5.00 log reduction after a 30 second exposure time.

Tested and Certified by: \_\_\_\_\_

\_\_\_\_\_  
Date